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ABSTRACT

The objective of this research program is to improve high temperature piezoelectric aluminum nitride (AlN) sensor technology to make it useful for instrumentation and health monitoring of current and future electrical power generation equipment. The program will extend the temperature range of the sensor from approximately 700°C to above 1000°C, and ultrasonic coupling to objects at these temperatures will be investigated and tailored for use with the sensor.

The chemical vapor deposition (CVD) AlN deposition process was successfully transferred from film production on tungsten carbide substrates to titanium alloy and silicon carbide (SiC) substrates. Further evaluation of the piezoelectric films on titanium caused it to be discarded as a candidate material due to an excessive thermal expansion coefficient mismatch, causing film failure upon reheating from room temperature. Deposition on SiC is proceeding well, with a highly conductive grade of silicon carbide required for practical use. Additional substrate materials, including refractory metals and conductive ceramics, have been considered but are generally not promising in light of the experience with titanium. Pulsed laser deposition (PLD) was investigated as an alternate means of creating the films as an alternative to CVD. A concurrent effort has focused on investigation of means of coupling ultrasound from the sensor into the test object at high temperature. A literature search combined with preliminary experimentation has resulted in the selection of two methods for coupling: low melting point glasses and metal foil- pressure couplant. The work in the next two years of the program will include continued improvement of the CVD deposition process, experimental testing of films and coupling at high temperatures, and a laboratory demonstration of the sensor in a simulated industrial application

TABLE OF CONTENTS

SECTION 1

INTRODUCTION

In order to use coal-fired electrical power generation equipment to its fullest potential, sensors are required which are capable of monitoring the "health" of critical system components. Such sensors can alert an operator to impending problems, if any, while reducing the need to shut down equipment for inspection. Benefits of health monitoring include reduced down time for inspection or unplanned outages. For equipment operating at high temperatures, this will also likely reduce thermal cycles and associated fatigue or wear. Equipment that can benefit from such health monitoring includes dynamic systems, such as turbine engines, as well as static systems such as gasifiers or other chemical processing facilities.

Past research conducted at the University of Dayton into the properties and behavior of Aluminum Nitride (AlN), a high temperature piezoelectric material, indicated that this material has a high potential for providing a basis for sensors which could be used for elevated temperature health monitoring, up to at least 1000°C (1800°F). The most straightforward implementation of AlN sensors is as ultrasonic transducers, for example as a thickness monitor in a critical region of a metal or ceramic component exposed to corrosive or oxidizing conditions. Ultrasonic thickness gages are widely available commercially for room temperature measurements. Ultrasonic waves may additionally be used to monitor bonds, such as that between a metal and a ceramic, as found in thermal and environmental barrier coatings commonly found in power generation equipment. Another possible implementation is as a high temperature accelerometer.

The first year of this three year effort focused on laying the groundwork for extending the sensor technology to the high temperature (>1000°C) regime. Highlights of the progress include reconstituting the process for depositing piezoelectric AlN films, investigating an alternative process, researching methods of coupling ultrasonic energy from the transducer to the test object at high temperature, and extending the process to deposit piezoelectric AlN films on substrates which can withstand the target temperature.

SECTION 2

EXECUTIVE SUMMARY

The goal of this project is to develop sensors that can be used at elevated temperatures, up to 1000°C, in power generation equipment. The primary target application for these sensors is equipment health monitoring. Historically, ultrasonic sensors have been used to evaluate properties of solid and fluid materials. Detected flaws may include cracks, voids, inclusions, porosity, or other discontinuities. Additionally, ultrasound is used in flowmeters, proximity sensors, and for material thickness measurements.

Conventional ultrasonic transducers and instrumentation are temperature limited because the piezoelectric materials used in the transducer cannot operate above their Curie point or depoling temperature. Operation above 100°C is considered "high temperature" with special requirements met by quartz or tourmaline transducers up to ~450°C, or complicated delay lines with complex cooling requirements (and usually) exposure times measured in seconds. Laser-based ultrasound and electromagnetic acoustic transducers (EMATs) are also used at high temperatures but are expensive or complicated. In addition, coupling the ultrasonic energy from the transducer into the test object becomes much more difficult above 100°C.

In the current project, ultrasonic transducers based on piezoelectric aluminum nitride (AlN) films are being developed which will enable the use of ultrasound for material evaluation at temperatures exceeding 1000°C. The polycrystalline AlN films are grown through a CVD process on a conductive substrate, which will then be incorporated into a completed transducer. The preexisting process for film development produced films on tungsten carbide substrates, and the current project is to evaluate alternative substrates capable of continuous operation at the 1000° target temperature.

The first alternative substrate attempted was titanium metal. Deposition on grade 5 titanium was unsuccessful; films were successfully deposited on grade 2 titanium. The unsuccessful attempts generally created a film, but the film disintegrated during cooling to room temperature at the end of the deposition. The process was advanced such that films on grade 2 titanium did survive the deposition process and subsequent polishing. These films produced ultrasonic energy of a strength equivalent to the films on tungsten carbide, but debonded upon reheating above room temperature. The conclusion was that the titanium is unsuitable as a substrate material because of the thermal expansion mismatch between the AlN film and substrate. A more promising substrate is silicon carbide, and two AlN films have been deposited on this material. The strength of their ultrasonic output currently is very weak but one survived heating to 400°C with no damage. Upcoming work will concentrate on further developing films on the silicon carbide and possibly additional substrate materials.

A literature survey on high temperature ultrasonic coupling has also been completed. There is no clearly defined best method for coupling at 1000°C, but two good candidate methods were found. The first is using molten glass as a couplant, with the glass chemistry chosen to match the desired operating temperature. A second method is to use a thin metal foil and high pressure to tightly couple the transducer and test object. These options will be experimentally evaluated during the second year of the program, both for ultrasonic efficiency and for compatibility with eventual target applications.

SECTION 3

EXPERIMENTAL

Piezoelectric AlN Film Deposition

Most of the efforts in the first year of the program were directed toward chemical vapor deposition (CVD) of piezoelectric aluminum nitride films. The thick films required for this project were created through a CVD process based on the deposition process described in Canadian Patents #2269581 and #2219404. This process begins with heating the substrate to ~700°C through induction heating in an inert environment in the CVD reactor. Argon, ammonia and aluminum bromide gases are flowed over the substrate, some of which react to form AlN on the surface of the substrate, at a deposition rate on the order of 0.01mm per hour. Prior to deposition, the surface of the substrate is polished and cleaned with a solvent. The process is essentially the same for all substrates tried to date.

During deposition, AlN crystals nucleate on the surface, and the crystals with the desired orientation for piezoelectric use grow in the thickness direction. Occasionally the crystal structure will be disrupted, leaving a pinhole gap or similar defect in the film; previous work with the deposition process has reduced the occurrence of such defects. More commonly, similar defects begin to form after the film has begun to grow, resulting in low areas in the films. The occurrence of defects in this manner limits the overall film thickness, as more defects occur as the film grows. The surface is initially polished by hand on 400 grit SiC paper after deposition to eliminate high spots for the initial ultrasonic evaluation. Further polishing on 400 and 600 grit paper is required to maximize the ultrasonic signal, with additional lapping sometimes used. Figure 1 shows a polished AlN film deposited on tungsten carbide.

Pulsed laser deposition (PLD) was also used to create three AlN films in order to evaluate it as an alternative method. In this process, an Al target is irradiated with the fundamental output (λ=1064 nm) of a Q-switched Nd:Yag laser with a heated substrate placed within a few mm of the irradiated area. The ablation is performed in a background pressure of ammonia in a vacuum chamber, forming AlN on the heated substrate. Placing the substrate at such a close distance to the target allows most of the ejected material to strike the target. The laser can deliver up to 400 mJ/pulse of its fundamental radiation. An infrared pyrometer was used to determine the substrate temperature during deposition. The films deposited by PLD have not been further polished. Figure 2 shows an AlN film deposited on silicon carbide using the PLD method.

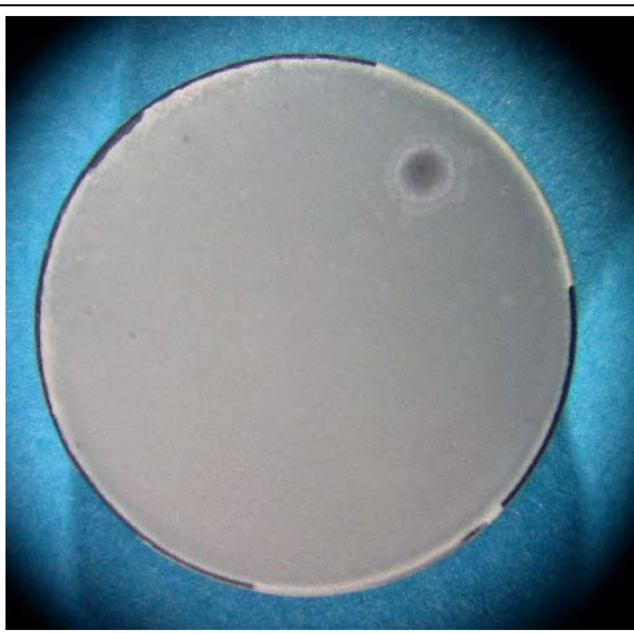

Figure 1. Polished AlN film on WC

Piezoelectric AlN Film Evaluation

One part of the film evaluation has been physical evaluation, primarily film adhesion to the substrates. The thin (<0.1 mm) AlN is not strong enough to be handled unsupported upon separation from a substrate. The CVD process was able to create well-bonded AlN films on tungsten carbide at the start of the project, but the bond strength on other substrate materials needed to be evaluated. The first test was simply time: some films had been known to delaminate during initial cooling while still in the CVD deposition reactor. Other films might delaminate in the following hours or days. This was the case with the films on Grade 5 titanium, so no further evaluation was possible.

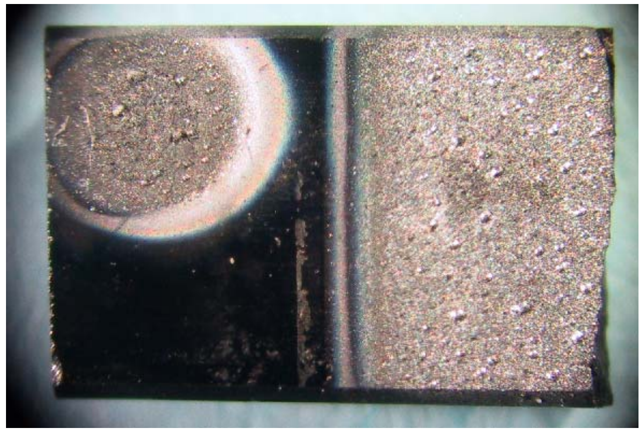

Figure 2. AlN film on SiC deposited by PLD.

If a film was stable, an initial polish as described above was performed prior to initial ultrasonics testing. Often films were received with a raised rim of AlN which was not well bonded, breaking off and leaving exposed substrate at the edge of the film. While not desirable, this was not fatal. However, some of these films would continue to delaminate with time, primarily on titanium. An additional test was to slowly heat the substrate using a hot plate. The films on tungsten carbide survived this as in the past, but no AlN films on titanium survived a single heating and cooling cycle, as shown in Figure 3. Future work will use a furnace to extend the heating range to the 1000° C target temperature.

Finally, testing for ultrasonic properties was performed, generally before a heating test. A Utex 340 ultrasonic square wave pulser/receiver instrument was used with an Agilent 54622A digital oscilloscope for these tests. The Utex 340 incorporates programmable low pass and high pass filtering, and can supply pulses of 5-80 ns with an amplitude of 100 to 500 V. Test voltages near 300 V were used for most films unless electrical shorting near damaged areas forced the use of lower voltages. For most testing, the film was placed on water-based ultrasonic couplant on a flat metal electrode. A two pronged probe from the pulser/receiver was then touched to this stack, with the ground on

Figure 3. AlN film on Ti delaminated by heating.

the metal electrode and the signal lead on the film's substrate. Pulse width, gain, and receiver filters were adjusted to maximize the signal on a film-to-film basis.

The frequency content of the first clear echo from the back of the substrate was used to determine the natural frequency of the film. The echo from the far surface of a 25.4 mm thick, 17-4PH stainless steel cylinder with parallel faces, used as the metal ground electrode, was also used to provide a film-to-film strength comparison without effects from the substrate geometry. Pulse width and gain settings were adjusted as needed. Averaging of multiple pulses was used to increase the signal to noise ratio. A typical ultrasonic waveform is shown in Figure 4.

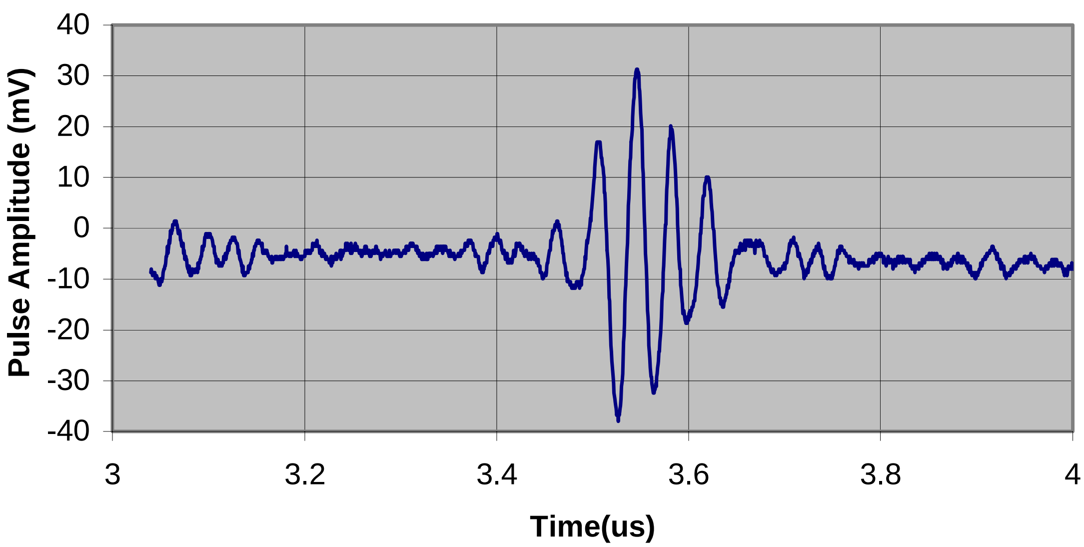

Figure 4. Typical ultrasonic pulse produced and received by AlN film.

SECTION 4

RESULTS AND DISCUSSION

Piezoelectric AlN Film Deposition

Task Description: The deposition of AIN films onto high temperature substrates will be studied and improvement made with respect to process and yield. Disposition of films will initially be done by an elevated temperature chemical vapor deposition process. An alternative deposition process, pulsed laser deposition (PLD), will also be attempted. Temperature of the substrate and PLD pressure will be varied to create an AIN film with piezoelectric behavior.

Subtask 1A: Conduct deposition of thick AIN film by chemical vapor deposition. Nov 2002-May 2005. This task is key to much of the rest of the project and is proceeding well. The CVD deposition apparatus was brought on-line and reliably produced piezoelectric AlN films on tungsten carbide substrates. Titanium substrates were next used and after a number of experiments with surface finish and several titanium alloys, AlN films were successfully deposited on a titanium substrate. Finally, films shown in Figure 5 were deposited on silicon carbide – a substrate not previously used by UDRI for AlN deposition. Additional film depositions will take place as the program proceeds.

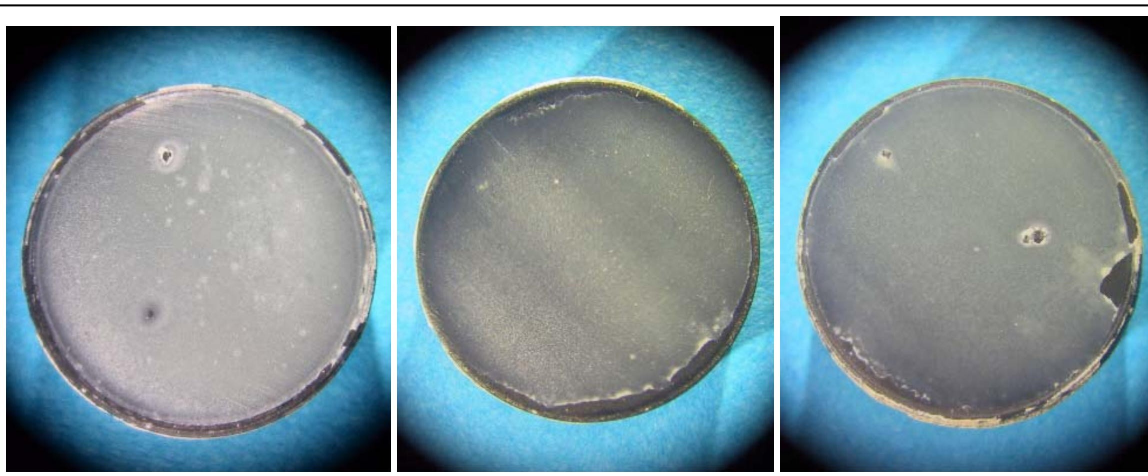

Figure 5. AlN films on SiC.

Subtask 1B: Identify the promising high temperature substrates with regard to thermal expansion, cost, machinability, and the ability to shape the substrate. Nov 2002-May 2004. An exhaustive search for alternate substrates has identified few candidate materials to date. Titanium metal seemed to be a good candidate because of its machinability, but had too great a difference in thermal expansion rates when reheated after deposition. Films would immediately separate from the substrate after heating to only a few hundred degrees Celsius. Silicon carbide (SiC) was identified as a good candidate for its thermal expansion and oxidation properties, but its electrical conductivity is borderline acceptable for strong piezoelectric operation of the AlN material. Additional materials, including several refractory metals (Mo, W), carbides (TiC), and conductive ceramics (titanium diboride, molybdenum disilicide) were rejected due to oxidation

sensitivity at temperature or excessive thermal expansion compared to the thermal expansion coefficient to the AlN films.
Analytical and experimental evaluation of additional candidate materials will continue at a low level until summer 2004. SiC appears to be the substrate of choice for the construction of the high temperature sensor, so efforts will focus on gathering more data on "conductive" grades of SiC currently available from various vendors. Where metal properties can generally be specified in terms of alloy content and heat treat, ceramics such as SiC are much more complex. Material properties such as conductivity vary based on processing method and the specific process used. For example, reaction bonded SiC actually contains a substantial amount of elemental metallic silicon, which affects the properties. SiC made by CVD is purer, more expensive, and available in more limited forms.

Subtask 1C: Experimentally explore the feasibility of AIN deposition onto identified new substrate materials such as graphite, alumina graphite, silicon carbide, titanium diboride, and molybdenum disilicide. January 2003-May 2005. As noted above, deposition was extended from the original tungsten carbide material to titanium metal and silicon carbide. Deposition on titanium was unsuccessful for Ti-6Al-4V (Grade 5) alloy, but was achieved with commercially pure (Grade 2) titanium; however, the resulting films were not stable when exposed to elevated temperatures. Initial results on silicon carbide, by contrast, are very encouraging. Deposition was achieved immediately without any process adjustments. Additional SiC depositions will be performed as substrates are selected as noted above.

Subtask 1D: Investigate the feasibility of using pulsed laser deposition of the AIN films onto substrates. The substrate pressure and temperature will be varied to determine conditions conducive to producing films specific for this high temperature sensor application. January 2003-January 2004. Three very small AlN films were produced on SiC substrates using the PLD method. The useful area of these films is likely too small for use in a full scale sensor, and difficulties have been encountered in evaluating these films. However, it is possible that this process might prove useful for "repairing" CVD films with pinhole or other defects. Additional work in this area will include more attempts to get an ultrasonic signal out of these samples and further analysis of the films. A PLD-produced film was shown in Figure 2.

Piezoelectric AlN Film Evaluation

Task Description: An evaluation of films deposited in task 1 will be completed in task 2. The films and high temperature substrates will be characterized structurally and ultrasonically. Characterization of the films will include the adhesion, oxidation, and continuity of the films when exposed to both room temperature and high temperature atmosphere. The ultrasonic properties of the films will also be determined by investigating the frequency, bandwidth, and signal strength at elevated temperatures. Using a scanning electron microscope with X-ray diffraction the surface and subsurface of the films will be analyzed to provide data on the crystal structure, defects, and bonding.

Subtask 2A: Characterize films by examining the thermal and physical properties of the films. Structural evaluation includes adhesion, oxidation, and continuity of the films as a function of time, temperatures up to 1000°C, and exposure to physical damage. December 2002- June

2005. Characterization to date has included baseline films on tungsten carbide and films on two new substrates, titanium and silicon carbide.
The films on titanium were found to be inferior upon heating above 100°C and not durable enough for deployment in a sensor. Heating causes films on titanium to disband from the surface of the substrate, rendering them useless as transducers. Surface roughening and additional cleaning to promote adhesion was ineffective. Most likely, the mismatch in thermal expansion between the titanium and AlN causes large stresses at the interface, breaking the bonds formed during deposition. While disbonded films on WC actually pulled away a small amount of the substrate material when they failed, the opposite effect was observed on the titanium: there was often a thin coating remaining on the titanium when the film disbonded. The poor results of titanium substrates pointed away from using other materials with a large expansion mismatch with the AlN, and toward lower expansion substrate materials.

The requirement for a low expansion material leads directly to ceramics, but electrically conductive ceramics are uncommon. One exception, silicon carbide, can be doped to be conductive and it was tried next. The SiC also has an acoustic impedance close to that of AlN, which should produce a high bandwidth ultrasonic transducer with very little ringing of the ultrasonic pulse. A preliminary evaluation of films on SiC is positive, with films remaining adhered through polishing and one film cycled to 400°C and back to room temperature with no ill effects. Ultrasonic data was not collected from these films, possibly due to the "low resistivity" of the chosen grade of SiC not being low enough. Additional evaluation of these films will be undertaken in the second year of the program.

Subtask 2B: Characterize films by evaluating the ultrasonic properties of the films and substrates. Ultrasonic evaluation includes frequency, bandwidth, and signal strength at ambient and elevated temperatures. December 2002- June 2005. New films on tungsten carbide show signal strength and properties similar to previously produced films. Films thick enough to have center frequencies below 30 MHz and substantial energy below 10 MHz are routinely produced. Films on titanium have similar ultrasonic properties with respect to center frequency and signal strength. Because the titanium is a closer acoustic impedance match to the AlN than the tungsten carbide substrates, the ultrasonic pulse shape is improved on the titanium, having a more broadband signal with less resonant ringing. Ultrasonic evaluation of the AlN films on silicon carbide have thus far been disappointing, with no detectable ultrasonic energy production. This will be more thoroughly investigated during the next reporting period. Ultrasonic data from AlN films on titanium and tungsten carbide is shown in Figures 6-9.

Subtask 2C: Characterize films using x-ray diffraction and scanning electron microscopy to examine the piezoelectric crystal structure, defects, bonding difficulties, and other material issues. March 2002-December 2004 . These evaluations have been postponed until AlN films which could be useful for eventual incorporation into a sensor are found to be mechanically and ultrasonically acceptable.

Subtask 2D: Relate substrate surface preparation to film quality (adhesion, defective areas, film thickness uniformity, and orientation of the piezoelectric crystals). December 2002-December 2004. Surface preparation has been noted, with a number of variations during the efforts to deposit on titanium. To date, minimal surface preparation of silicon carbide substrates has been required, with apparently good films resulting from simply polishing and cleaning the as-received substrate material.

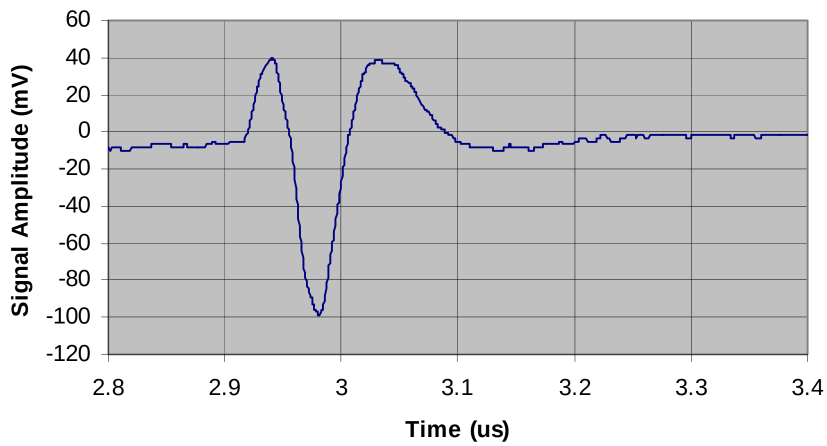

Figure 6. First back-surface echo of ultrasonic pulse from ~13 MHz AlN film on WC.

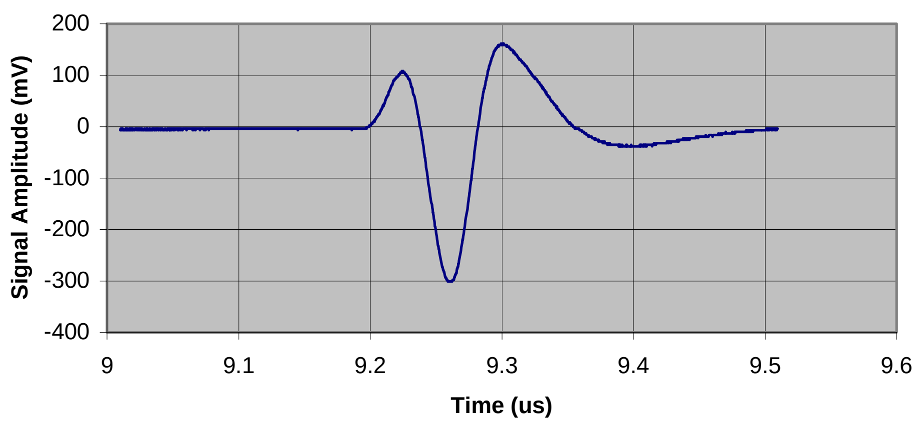

Figure 7. First echo in steel test block of ultrasonic pulse from ~13 MHz AlN film on WC.

High Temperature Coupling

Task Description: A literature survey will be made of methods to conduct ultrasonic coupling at elevated temperatures and those that are most suited to equipment health monitoring. Those methods that appear most suited for the application will be verified in the laboratory, in particular, the effect of the coupling layer on the impedance matching will be examined.

Subtask 3A Perform literature survey re high temperature coupling. January 2003-August 2003. A literature survey was completed by UDRI with the help of a mechanical engineering student employed as part of our cooperative education program. Commercial products and a variety of historical coupling methods were identified, including molten glasses, ceramic adhesives, and metal foils. The data gathered will be further evaluated and one or more of the most promising methods for use with AlN transducers will be selected.

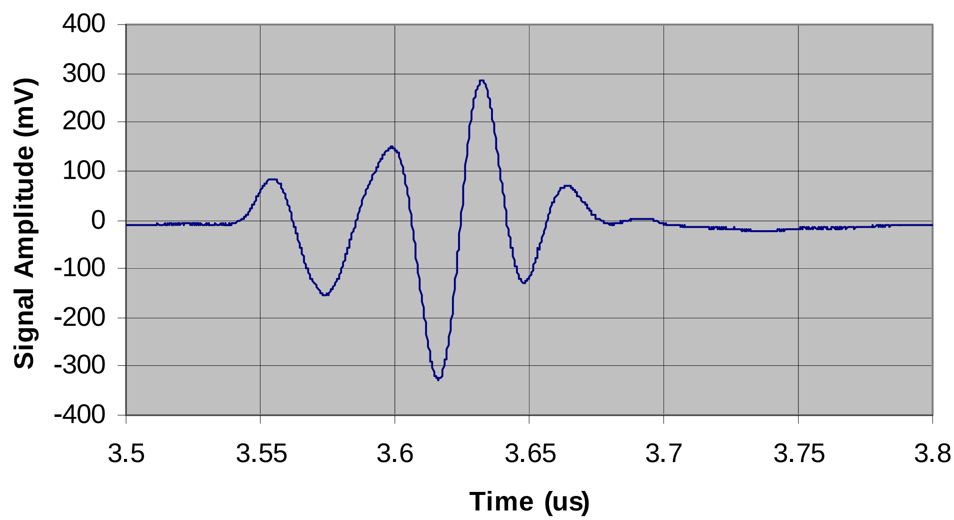

Figure 8. First back-surface echo of ultrasonic pulse from ~30 MHz AlN film on titanium.

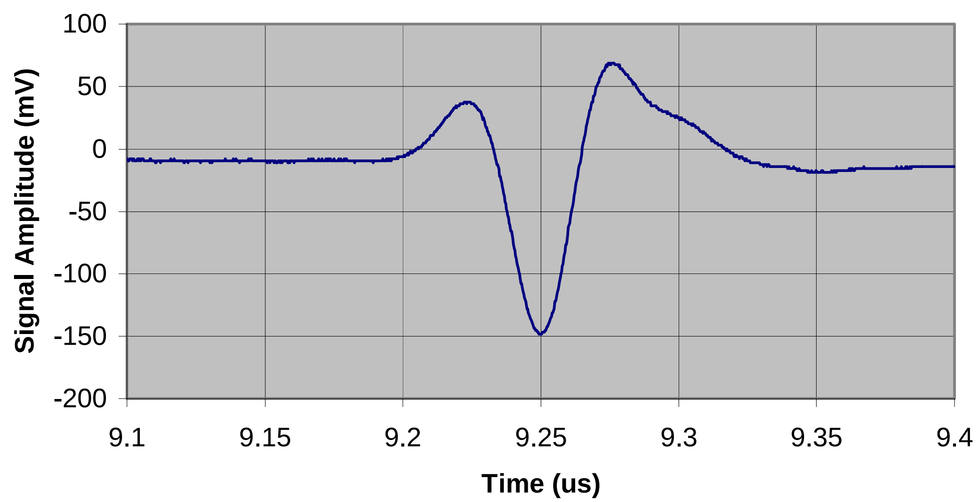

Figure 9. First echo in steel test block of ultrasonic pulse from ~30 MHz AlN film on titanium, with higher frequencies filtered out by test block

A coupling material is required to transmit ultrasonic energy from the transducer into the test object, displacing air between the transducer and the surface of the test object that would cause the sound energy to be reflected. The reflection of sound energy is caused by a mismatch in the compressibility of two materials, as measured by the acoustic impedance. Air has a dramatically lower impedance than solids or liquids, and the higher the degree of an impedance mismatch the more sound is reflected. Therefore, only a very small fraction of the ultrasonic energy from the transducer is transmitted to the test object, and a similar percentage of the returning sound energy is not received by the transducer.

To reduce this signal attenuation many different methods and couplants have been employed. At room temperature, typical couplants include water, glycerin, elastomers and various kinds of specially produced gel. At high temperatures the number of available couplants rapidly decreases. Specially produced commercial couplants can only reach a temperature of 590° C. However, some applications require temperatures exceeding 800° C.

Several methods for coupling ultrasound at very high temperatures have been attempted. The first and possibly the least plausible is the Newton's rings method. This method entails polishing mating surfaces flat and parallel such that the air gap between them is reduced below 0.01 μm. Newton's rings optical method would then be used to evaluate the flatness of the surfaces. However, even with an air gap of 0.001 μm. only ultrasonic with frequencies less than 5 MHz would be transmitted.

More conceivable is the use of a liquid or solid couplant. Metal foils such as zinc are sometimes used for couplant at lower temperatures. However, at the 800-1000°C temperatures required for this research, zinc would easily melt, flow away, and oxidize. A metal with a much higher melting temperature than 800° C would be much more effective. Platinum, for instance, is often used up to 1000° C, making it an excellent candidate for a couplant. If thin foils were used in small areas, especially for semi-permanent transducer installations, the expense of platinum would be offset by the very small quantities required.

A better solution to this problem is most likely a form of glass. Research has turned up an article stating that coupling temperatures nearing 800 degrees Celsius had been reached by using a form of glass solder. Glass, unlike liquid metals, will most likely be able to adhere to both surfaces, preventing air pockets from blocking the transmission of ultrasound. However, there are an exceedingly high number of chemical compositions for glass and thus a great deal of research must be done into what will allow for the best sound transmission. An additional unknown is if the molten glass layer will be thin enough to have a minimal effect on the strength of the electric field through the thickness of the AlN film.

The next step of this investigation will be to develop a method for testing the glass couplants and possibly the platinum or other metal foil to determine what performs the best at the required temperatures. Research into how the chemical compositions of glass affect sound transmission will also be necessary.

Subtask 3B Design high temperature apparatus for testing films and coupling to test objects. August 2003-December 2003. Based on the literature survey, the technical community appears to have had limited success in developing ultrasonic coupling methods suitable for use at high temperatures. Accordingly, this subtask will be expanded through the second year of the program, and will form the basis of an undergraduate Honors Thesis for the student who performed the literature survey. Although the task lists designing high temperature apparatus, it will actually include design, construction of apparatus, and using it to test ultrasonic coupling at high temperature.

SECTION 5

CONCLUSION

The project is on schedule to complete the desired tasks within the program period and budget. The first year's expenditures have been less than 30% of the project total, but future work, especially the demonstration project, will increase the rate of expenditures. In addition, at least two students will be employed this coming summer as more experimental work is required. Selection of a demonstration project application is an important task in the coming year.

Piezoelectric AlN film deposition is proceeding well. The work to date has resulted in the identification of a very small number of suitable substrate materials; SiC may be the only likely candidate substrate to extend the sensor's capabilities to the desired 1000°C range. The sensitivity to thermal expansion coefficient eliminates virtually all metals, which in turn reduces the likelihood that sensors could eventually be deposited directly on metallic components. Pulsed laser deposition appears to be impractical at this time, but the existing PLD films will be further examined to verify that the AlN produced actually has the required piezoelectric structure.

In addition to the CVD films, a possible future source of piezoelectric AlN has been identified. A technology start-up company called Crystal IS, located in New York, has developed the technology to grow large single crystals of AlN in a boule form for use in experimental semiconductors. This material may also be useful as a transducer material. No material is available as of August 2003, but there is a possibility that some might become available for purchase during this program. If so, a sample will be procured for comparison with the CVD films, and for evaluation for sensor applications.

Research to date has not been complete enough to be submitted to conference proceedings or publication, but plans to do so include: (1) A student thesis and associated presentation regarding high temperature ultrasonic coupling. (2) A conference paper on the high temperature ultrasonic transducer advancements during the program is anticipated in 2005; the likely venue will be the annual Review of Progress in Quantitative Nondestructive Evaluation (QNDE). (3) A conference paper or journal article is also planned regarding the application of the final sensor design to the selected demonstration application.

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